



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification 5 :</b> B01J 13/22, 20/32, 2/16 B01J 2/30	<b>A1</b>	<b>(11) International Publication Number:</b> WO 93/22048 <b>(43) International Publication Date:</b> 11 November 1993 (11.11.93)
<b>(21) International Application Number:</b> PCT/SE93/00379 <b>(22) International Filing Date:</b> 29 April 1993 (29.04.93)  <b>(30) Priority data:</b> 9201349-9                      29 April 1992 (29.04.92)                      SE  <b>(71)(72) Applicants and Inventors:</b> ELLERS, Berne, F. [SE/SE]; Diabasvägen 7A, S-269 41 östra Karup (SE). APPEL- GREN, Curt, H. [SE/SE]; Benjaminssons väg, S-434 91 Kungsbacka (SE).  <b>(74) Agent:</b> INGER, Lars, Ulf, Bosson; L&U Inger Patentbyrå AB, Garvaregatan 12, S-262 63 Ängelholm (SE).		<b>(81) Designated States:</b> AU, BR, CA, FI, HU, JP, KR, NO, PL, RU, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> ABSORBING MATERIAL  <b>(57) Abstract</b>  The present invention relates to a process for preparing an absorbing material comprising a polymer material which swells and forms a gel while absorbing a liquid, whereby the absorbing material has been provided with a meltable, adhesive structure forming composition.		

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	MR	Mauritania
AU	Australia	GA	Gabon	MW	Malawi
BB	Barbados	GB	United Kingdom	NL	Netherlands
BE	Belgium	GN	Guinea	NO	Norway
BF	Burkina Faso	GR	Greece	NZ	New Zealand
BG	Bulgaria	HU	Hungary	PL	Poland
BJ	Benin	IE	Ireland	PT	Portugal
BR	Brazil	IT	Italy	RO	Romania
CA	Canada	JP	Japan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SK	Slovak Republic
CI	Côte d'Ivoire	LI	Liechtenstein	SN	Senegal
CM	Cameroon	LK	Sri Lanka	SU	Soviet Union
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	MC	Monaco	TG	Togo
DE	Germany	MG	Madagascar	UA	Ukraine
DK	Denmark	ML	Mali	US	United States of America
ES	Spain	MN	Mongolia	VN	Viet Nam
FI	Finland				

ABSORBING MATERIAL.DESCRIPTIONTechnical field

5 The present invention relates to a process for manufacturing a water absorbent article in which particles of a water swellable polymer are fixed to a substrate absorbing material.

10 The object of the present invention is to obtain absorbing products, preferably water-absorbing products, which can be applied to a fibrous structure in a safe way.

Background of the invention.

15 Absorbing products, and in particular so called super absorbing products, are products and compounds that absorb typically 40 to 60 times its weight with respect to a saline solution and 100 to 200 times its weight with respect to pure water. The products are used within different sectors, but have preferably been used within the food industry sector, hygienic products sector, and medical technical sector. In the first mentioned case for the  
20 absorption of liquid from prepacked foodstuffs such as meat and fish, for example to absorb blood juice or melt water from iced fish, or in the second case with regard to hygienic articles of manufacture as diapers and sanitary napkins for the absorption of urine. These super absorbents are generally present in powdered form which means that when being introduced into a substrate, fibrous structure of cellulose or synthetic fibres or mix-  
25 tures thereof, which is quite common in hygienic products, these absorbents will be readily released from the structure during transport, and will fall out with an accompanying reduced absorbing effect as a consequence. One has tried to eliminate this  
30 by compressing the structure maximally in order to bind the the absorbent but has not succeeded completely. Other ways of solving the problem is to manufacture laminates where the absorbent is distributed between two layers of materials which are bound  
35 together using a thermoplastic powder. The absorbent can also be manufactured in the form of a polyacrylate fibre being coated with a high absorbing coating, but this will increase costs of

the product considerably. As one, in the hygienic products of today, such as in a diaper, adds the absorbent intermittently in islands in an attempt to optimize the efficiency thereof, this will create a problem in that so called gel blocking easily occurs which means that all particles of the absorbent can not be reached by a liquid but remain unused. The technology of today further includes a limitation in that different qualities of absorbents and/or sizes of particles of absorbent can not be used together in a simple way.

10

A further problem using the absorbents of today in the form they are used is that only certain particle sizes can be used and then preferably the larger particle sizes. The smallest particles can not, due to a the off-separation mentioned above be used which means increased costs.

15

At the formation of the final products generally dry methods are used as the presence of moisture means that the absorbents are being activated. Such dry methods are used at the manufacture of diapers, sanitary napkins, non-woven material for hygienic and medical use, filter materials, package materials, whereby soft paper, so called fluffy pulp of cellulose fibres and staple fibres of polyethylene, polypropylene, polyesters, polyamides, or other poly olefinic fibres, is used. The selection of fibre depends on the different demands which are requested from the final product. The differences in the types of fibres are primarily in the specific area they provides. The advantage of so called poly olefinic fibre is that it has a large specific area, 8 to 15 m<sup>2</sup>/g thus encapsulates a powder better in the capillary structure but as the same fibre requests high temperatures for being activated the cellulose fibre will easily become discoloured and may even be ignited. Staple fibre, specific area 2 to 3 m<sup>2</sup>/g has a very poor capability of binding the absorbent particles in a non-woven material.

30  
35

Fluffy pulp is most often formatted on a tissue of cellulose in order to prevent losses of short cellulose fibres. In diapers

and sanitary napkins a surface layer of non-woven material, such as of polypropylene, is most often used. In order to maintain the absorbent the request for density of these materials has been increased which in turn render passage of liquid of the surface layer more difficult.

As evident from above great problems exist in the off-separation of absorbent from the material.

There are thus a number of problems which look for their solutions.

#### Description of the present invention

It has now surprisingly been shown possible to be able to solve these problems by means of the present invention which is characterized in that the absorbing material is provided with a structure of adhesive material and in particular the process of the invention is characterized in that

- a) applying to the surface of water swellable particles a structure of a meltable adhesive composition to form secondary particles, the meltable, adhesive composition being applied in molten, non-solvent containing form to said particles,
- b) bringing the secondary particles into contact with said substrate and
- c) applying heat to melt the meltable, adhesive composition of the said secondary particles to cause said secondary particles to adhere to said substrate.

Further characteristics are evident from the accompanying claims.

By means of the present invention there is provided (a) that the absorbent can be bound to a substrate, either completely or in fibre form by means of the meltable, adhesive composition, (b) that the absorbent can be bound to a fibre web while being formed in dry condition, (c) that the absorbent maintains its absorbing ability and (d) that different particle sizes of the

absorbent as well as different absorbent qualities can be bound and used together. Further advantages are evident from the following.

- 5 Absorbing material hereby intends to mean polyacrylates, carboxymethyl cellulose, modified cross-linked starch, and other polysaccharides. Such absorbents are sold under e.g. the trade names SALSORB 84, and SALSORB DD (polysodiumacrylate), AQUA KEEP 10 SH-P, AQUA KEEP X 50, AQUA KEEP X 65, LANSEAL-F (polyacrylate 10 fibre), DRYTECH 510 (cross-linked acrylate copolymer), GELOK 40 (cross-linked starch grafted polyacrylate polymer), AKUCCELL SW (cross-linked sodium carboxymethyl cellulose).

- Suitable structure forming materials to provide the meltable, 15 adhesive composition are polyols, polyethylene glycol, emulsifiers in the form of esters (Dimodan, Dimodan TH; distilled monoglycerides, Acidan; citric acid esters of monoglycerides, Cetodan; acetic acid esters of monoglycerides, Lactodan; lactic acid esters of monoglycerides, Panodan; diacetyl tartaric acid 20 ester of monoglycerides, Promodan; propylenglycol esters of fatty acids, Triodan; polyglycerol esters of fatty acids), fatty acid alcohols (cetanol, stearyl alcohol, palmitol), fatty acids (stearic acid, palmitinic acid), hardened fats, waxes (bees wax, bamboo leaf wax, carnauba wax, Japan wax, shellac wax, cotton 25 wax, micro-crystalline wax), paraffin wax, melt glue (Lunatack based on colophonium resins), INSTANT-LOK, either used alone or in different combinations thereof.

- The structure forming material may be provided with up to 30 % 30 by weight of a plasticiser in the form of glycerin, diethylphtalate, Citroflex esters of citric acid), triacetin, propylene glycol, or sorbitane fatty acid esters, such as sorbitane mono-laurate, sorbitane monostearate, sorbitan monopalmitate, and sorbitane sesqui oleate, sorbitan monooleate, as well.

- 35 Different structure forming materials can be combined, as well as different plasticisers. Hereby a slower softening temperature

is achieved, which is positive contrary to a very rapid, exact melting point.

The term structure forming material used means that the material does not form a continuous coating onto the waterswellable particles, but form either a net structure, or a spotwise appearing structure which is able to form a mechanical bond between the absorbent and the substrate used as a carrier to said absorbent.

10 The structure of meltable material should have a melting temperature exceeding the temperature of use with about  $10^{\circ}\text{C}$ , i.e. in case of use in diapers and sanitary napkins, a melting temperature of at least  $50^{\circ}\text{C}$ .

15 To obtain an improved water penetration at the use of water insoluble structure forming materials water soluble crystals can be incorporated into the structure.

Other substances that may be incorporated into the structure are odour eliminating substances in the form of calcium hydrogen phosphate having 2 crystals of water, flavour providing compounds (deodorants), bacteriostatic or bacteriocidal compounds such as iodine, sorbic acid, benzoic acid, hexachlorophen, lactic acid.

25

Further examples of substances that might be incorporated are fertilizers.

The amount of structure forming material onto the absorbent should amount to 5 % by weight.

The absorbent having a particle size of from  $<5\text{ }\mu\text{m}$  to up to 1 mm, typically 90 to 8000  $\mu\text{m}$ , can be provided with the structure forming material in a coating pan (pan-coating), drum coating, roto-coater, intense mixer, so called batch mixer, fluid bed coater, or using a melt application technology disclosed in SE-A-8500487-7.



At the application in an intense mixer the absorbent and the structure forming materials are brought and treated together during heat addition. At the application in a fluid bed device  
5 the absorbent is introduced into the device and is fluidized using air. The structure forming material can then be added as a solid product and applied to the absorbent while heated, or be added as a melt through nozzles provided above or underneath the fluidized bed.

10

The application of the solid product with itself can be done in an apparatus described in SE-C-7903053-2 and in such a way as described in SE-A-8500487-7.

15 The apparatus according to SE-C-7903053-2 comprises a mixing house provided with a cover. Within the housing a disc is arranged which disc consists of an upper disc and a lower disc, which disc is rotatably mounted around a shaft by means of a bearing. Between the upper and the lower discs there is a hollow  
20 space which is connected to the upper side of the upper disc via an annular slot consisting of two parts which both take the shape of a circumferential surface of a cut cone with its point directed downwardly. The upper disc has blades cut on its upper side and optionally cut, or mounted blades on its side under-  
25 neath. Between the blades spaces exist which are intended to receive particles to be coated while said space optionally being divided into compartments by means of said blades is intended to receive the melt which is to be used for coating. The lower disc is provided with an edge extending around it close to the open-  
30 ing of the slot. At its periphery the lower disc is provided with blades for throwing out the final product via a diffusor and an outlet. The cover is provided with a compartment placed above the centre of the disc which compartment is arranged to receive the particulate solid material for a further transport  
35 thereof to said above mentioned spaces between the blades. The particulate solid material is fed to the compartment of the cover by means of a transporting device, e.g. a feeding screw. The



liquid phase is being fed to the space via a tube.

At the application operation according to SE-A-8700487-7 the discs are rotated within the housing around the shaft with a revolving speed of between 1000 to 500 rpm. A suitable peripheral speed using a disc diameter of 300 mm is 1500 to 5000 m per minute whereby the lower speed is used for embedment/agglomeration and the higher for coating. The coating melt is thereby fed to the apparatus via the tube to the hollow space between the upper and the lower discs. The tube is thereby provided with heating means in order to keep the material in melted, liquid form. By means of the centripetal force and the blades the melt is thrown out-wardly through the slot and further through the outer slot. The melt hereby takes the form of a film which extends outwardly all the time simultaneously as it becomes thinner. When the melt leaves the outer slot the film is torn up into very small droplets when it leaves the edge, whereby a mist curtain of droplets having a microscopic size are formed. Simultaneously with the addition of melt through the tube a particulate material is added by means of the transporting device to the compartments and further on to the spaces on the upper side of the upper disc. From there the particulate material is thrown, by means of the centripetal force, and the blades, outwardly towards the periphery of the upper disc, simultaneously as it is deagglomerated into primary particles which meet with and pass the mist curtain at the edge. The particles hereby obtain a surface coating of the melt and are further thrown outwardly to the blades arranged at the periphery of the lower disc, which blades throw the product out off the apparatus via the diffusor and the outlet. Depending on the number of blades their height and the throughput, which can be varied using the outlet area, one can get an agglomeration of the particles, or get the particles out as separate sole, coated particles. The granulate obtained is directly chilled with air during the throwing out thereof and is further chilled at a following separation in a cyclone or the like.

The invention will be further disclosed in the following with

reference to some embodying examples.

Example 1

5 A 50:50 mixture of a slow absorbent, Aqua Keep X65, and a rapid absorbent, Aqua Keep 10 SH-P (acrylic acid polymer) was provided with 23 % calculated on the final weight, with a structure forming material consisting of 25 % of Dimodan TH, 50 % of polyethylene glycol (PEG 6000/PEG 400 90:10), and 25 % of Lunatack (melting glue) according to the process according to SE-A-10 8500487-7.

Example 2

15 A 50:50 mixture according to Example 1, was provided with 20 % by weight of the final weight, with a structure forming material consisting of 75% of Dimodan (an emulsifier), and 25% of Cetodan (an emuldsifier) according to the same process as in Example 1.

Example 3

20 A 50:50 mixture according to Example 1, was provided with 20 % by weight of the final weight, with a structure forming material consisting of 75% of Dimodan TH, and 25% of Cetodan according to the same process as in Example 1.

Example 4

25 A 50:50 mixture according to Example 1, was provided with 20 % by weight of the final weight, with a structure forming material consisting of polyethylene glycol (PEG 6000/PEG 400 90:10) according to the same process as in Example 1.

30 The products thus obtained were tested with regard to adhesive-ness to a dry formulated web having a layer of staple fibre surrounded on each side by a layer of tissue fibre (non-woven, cellulose fibre). The granular grains provided with a meltable structure forming material and having a size of 500 to 1000  $\mu$ m  
35 were adhered using heat radiation for 20 sec. At shaking no off-separation could be observed.

The products were tested with regard to absorbent activity as well, in a 0.9 % saline solution. Hereby 1 g of the respective granular powder was dissolved in 70 ml saline solution for 5 min whereupon the gel obtained was allowed to drip off placed on a screen for 2 min., whereupon the volumes obtained were measured. Then the gel was placed between two pieces of filter paper the weights of which were determined prior to and after 60 sec. The ability of the filter paper to absorb liquid from the gel is a measure of the so called rewetting degree of the gel. The following data were obtained, Table 1.

TABLE 1

Sample	Volume absorbed (ml)	Time for stiffening (sec)	Rewetting degree (g)	Revised absorption (ml)
X65	58.1	165	2.504	
10 SH-P	69.5	12	2.463	
Ex. 1	54.5	120	2.57	68.1
Ex. 2	55.8	45	2.565	69.8
Ex. 3	55.8	60	2.585	69.8
Ex. 4	54	120	2.535	67.5

1 g of powder contains 0.8 g of absorbent. The amount of liquid absorbed is direct proportional to the weight of absorbent. Revised values with regard to weight are thus given. It is evident from these that complete absorption is obtained in spite of the applied structure forming material. In the same way the slow absorbent is thus influenced that the combination becomes faster in Ex. 2 and Ex. 3 than can be expected.

Besides the above indicated areas of use one can utilize the present invention for the binding of grains/seeds in a non-woven structure. Thus it is possible to bind grass seeds in a simple way together with an absorbent and then roll it out for providing of a lawn.

CLAIMS.

1. A process for manufacturing a water absorbent article in which particles of of a water swellable polymer are fixed to a substrate, which process comprises:
  - 5 a) applying to the surface of water swellable particles a structure of a meltable adhesive composition to form secondary particles, the meltable, adhesive composition being applied in molten, non-solvent containing form to said particles,
  - b) bringing the secondary particles into contact with said substrate and
  - 10 c) applying heat to melt the meltable, adhesive composition of the said secondary particles to cause said secondary particles to adhere to said substrate.
- 15 2. Process according to claim 1, characterized in that the meltable, adhesive composition is at least 5 % by weight of the final product.
3. Process according to claim 1, characterized in that the absorbing material is polyacrylates, carboxymethyl cellulose, modified cross-linked starch and other polysaccharides.
- 20 4. Process according to claim 1, characterized in that the meltable, adhesive structure forming material is a polyol, polyethylene glycol, emulsifiers in the form of esters, fatty alcohols, fatty acids, waxes, paraffin waxes, melting glue, and combinations thereof
- 25 5. Process according to one or more of the preceding claims, characterized in that the structure forming material further comprises a plasticiser in the form of glycerine, diethylphtalate, Citroflex, triacetin, propylen glycol, and combinations thereof.
- 30 6. Process according to one or more of the preceding claims, characterized in that the structure forming material further comprises odour eliminating compounds, flavour providing substances (deodorants), bacteriostatic or bacteriocidal compounds,
- 35

and combinations thereof.

7. The use of an absorbing material prepared in accordance with claim 1 to 6, in a product intended for food-stuffs.

5

8. The use of an absorbing material prepared in accordance with claim 1 to 6, in a product intended for hygienic purposes.

9. The use of an absorbing material prepared in accordance with  
10 claim 1 to 6, in medical technical product.

10. The use of an absorbing material prepared in accordance with claim 1 to 6, in technical horticultural product.

15

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 93/00379

## A. CLASSIFICATION OF SUBJECT MATTER

IPC5: B01J 13/22, B01J 20/32, B01J 2/16, B01J 2/30  
According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US, A, 4675236 (MASAAKI OHKAWARA ET AL), 23 June 1987 (23.06.87), column 2, line 57 - column 5, line 9 --	1-10
A	EP, A1, 0330331 (CHISSO CORPORATION), 30 August 1989 (30.08.89), page 3, line 49 - page 4, line 54 --	1-10
A	DE, A, 2138037 (INNOVA CORP.), 8 February 1973 (08.02.73), claims 13-16 -- -----	1-10

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

## \* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

28 June 1993

Date of mailing of the international search report

21 -07- 1993

Name and mailing address of the ISA/  
Swedish Patent Office  
Box 5055, S-102 42 STOCKHOLM  
Facsimile No. +46 8 666 02 86

Authorized officer

Eva Iversen Hasselrot  
Telephone No. +46 8 782 25 00



## INTERNATIONAL SEARCH REPORT

Information on patent family members

28/05/93

International application No.

PCT/SE 93/00379

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 4675236	23/06/87	EP-A- 0234078 SE-T3- 0234078	02/09/87
EP-A1- 0330331	30/08/89	AU-A- 3000889 DE-U- 6890154 JP-A- 1215783 JP-B- 4069598 US-A- 5147442	24/08/89 25/06/92 29/08/89 06/11/92 15/09/92
DE-A- 2138037	08/02/73	NONE	